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(54) Title: NOVEL POLYPROPYLENE COMPOSITIONS (57) Abstract Novel high melt flow polypropylene compositions useful in the manufacture of fibres and films are disclosed. The novel polypropylene compositions contain effective stabilizing amounts of selected N,N-dialkylhydroxylamines and organic phosphites or phosphonites which provide for a reduction in the amount of smoke, oil and wax generated when such compositions are spun or extruded into fibers or films.		

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NOVEL POLYPROPYLENE COMPOSITIONS

This invention relates to polypropylene compositions useful in the manufacture of fibers and films. In particular, the invention relates to polypropylene compositions containing selected additives which provide for reduced devolatilization as evidenced by a reduction in the amount of smoke, oil and wax generated when such compositions are spun or extruded into fibers or films.

BACKGROUND OF THE INVENTION

Because of their superior physical properties and ease of processing, certain higher melt flow polypropylene resins have found widespread use in the manufacture of fibers and films. Such high melt flow polypropylene resins have been found to be particularly useful in processes involving melt extrusion of fine fibers such as those produced in the "spunbond" process and in the manufacture of thin films. However, a major disadvantage in the use of these higher melt flow polypropylene resins in processes where the extrusion temperatures can range as high as 280°C or higher is the evolution of volatiles which condense leading to a build-up of oil or waxy deposits on the extrusion equipment requiring frequent cleaning and the formation of aerosols in the area surrounding the extrusion equipment creating an undesirable atmospheric condition known in the art as "smoke". While this problem has long been recognized in the art, recent developments including greater environmental awareness and concerns for employee safety as well as increased demands for production efficiencies has heightened the need for an economically acceptable solution.

Considerable effort has been directed to solving the smoke problem by reducing the amount of low molecular weight oligomers formed in the polymerization reactor during production of the polypropylene resins and during the cracking of such resins where

necessary to obtain higher melt flow products. For example, USP 5,703,203 suggests that the presence of oligomers in polypropylene polymers causes the emission of smoke during melt extrusion and proposes a method of stripping oligomers from the resins before extrusion. This approach has not provided a completely satisfactory solution to the problem of reducing smoke because while the resin being fed to the fiber or film extrusion equipment may contain smaller amounts of oligomers, new oligomers and other lower molecular weight volatiles are formed as a result of thermal oxidation reactions during the high temperature extrusion process which devolatilize and contribute to the formation of smoke and oily or waxy build-ups on the extrusion equipment.

It has been suggested in the prior art that the presence of antioxidants in polyethylene resins and low melt flow polypropylene resins can reduce the emission of volatiles resulting from thermaloxidation reactions which occur at the high temperatures involved in industrial extrusion processes. In an article titled "Effectiveness of Antioxidants: Suppression of Evolution of Gaseous Degradation Products from Low-Density Polyethylene During Thermo-Oxidation", Madsen et al, Polymer Degradation and Stability, Elsevier Applied Science Publishers Ltd., England, 12 (1985), pgs. 131 to 140, the authors evaluated the effect of a number of commercially available antioxidants in suppressing the emission of volatiles produced during thermal degradation of polyethylene. In an article titled "Thermal Oxidation of Polypropylene in the Temperature Range of 120-280°C", Hoff et al, Journal of Applied Polymer Science, Vol. 29, pgs. 465-480 (1984), the authors demonstrate that the presence of large amounts of commercially available antioxidants in low melt flow polypropylene resins can suppress the emission of certain low molecular weight volatiles resulting from thermal degradation at high temperatures. The authors also found that the suppression of acetone was enhanced by combining an organic phosphite additive with the antioxidant. Neither of these articles suggests using antioxidants or other additives to suppress the emission of volatiles during the extrusion of high melt

flow resins and in fact applicants have found in their work that the use of such commercially available antioxidants alone or in combination with organic phosphite additives in high melt flow resins does not provide an adequate reduction in smoke when such resins are extruded at high temperatures.

It is well known that polypropylene without the benefit of protective additives will degrade during processing and longer term exposure to sunlight and the atmosphere. Various classes of chemical compounds are recognized as being useful to protect and improve the performance of polypropylene by functioning, for example, as antioxidants, process stabilizers, acid acceptors, lubricants, nucleating agents and ultraviolet stabilizers. However, other than the suggestion in the articles discussed above that certain antioxidants alone or in combination with organic phosphites may suppress the emission of certain volatiles during high temperature degradation of low melt flow polypropylene, the art has not recognized the potential benefit of using selective additives to suppress the emission of volatiles and thus reduce the presence of smoke during melt extrusion of high melt flow polypropylene resins.

N,N-dialkylhydroxylamines are recognized in the art as being useful process stabilizers for polyolefins including polypropylene. See for example USPs 4,590,231, 4,876,300 and WO 94/24344. USP 4,876,300 suggests that there are advantages in the use of long chain N,N-dialkylhydroxylamines as process stabilizers for polyolefins and teaches the use of such hydroxylamines in combination with other additives including organic phosphites in stabilizing polyolefins including low melt flow polypropylene. WO 94/24344 teaches a three component system for stabilizing polypropylene which includes a long chain N,N-dialkylhydroxylamine, an organic phosphite and a hindered amine. This publication suggests that the hindered amine can replace conventionally used phenolic compounds to provide long term heat aging stability and that the three component system provides excellent resistance to gas fading. None of these patents suggest using the hydroxylamines alone or in combination with other additives such as

organic phosphites to reduce the formation of smoke during melt extrusion of high melt flow polypropylene resins.

SUMMARY OF THE INVENTION

The present invention relates to high melt flow polypropylene compositions which provide for reduced smoke formation when extruded into fibers or films. The inventive composition comprises a high melt flow polypropylene resin, a stabilizing amount of an N,N-dialkylhydroxylamine and a stabilizing amount of an organic phosphite or phosphonite and, optionally, other conventional additives such as antioxidants, acid acceptors, light stabilizers, ultraviolet light absorbers and the like.

DETAILED DISCLOSURE

The novel polypropylene compositions of the present invention comprise a high melt flow polypropylene resin, a stabilizing amount of an N,N-dialkylhydroxylamine and a stabilizing amount of an organic phosphite or phosphonite and, optionally, other conventional additives.

High melt flow polypropylene resins useful in the composition of the present invention can be homopolymers of propylene, random copolymers of propylene with ethylene or higher alpha-olefins such as butene containing at least 70% by weight (w) preferably at least 80% w propylene, and polypropylene impact copolymers. The homopolymer phase of such impact copolymers is preferable a polypropylene homopolymer but may contain up to 5% w of a comonomer such as ethylene or a higher alpha-olefin. The rubber phase of the impact copolymer is a copolymer of ethylene and propylene with an ethylene content of between 30% w and 90% w, preferable 45% w to 75% w. The amount by weight of rubber phase present in the impact copolymer ranges between 5% w to 50% w and preferably from about 10% w and 35% w.

The high melt flow polypropylene resins useful in the present invention have a melt flow of at least 20 dg/min (as determined by ASTM D-1238, Cond. L), and preferably in the range of 30 to 3000 dg/min and a narrow molecular weight distribution Q in the range of about 2 to about 6 where Q is defined as the gel permeation chromatography (GPC) parameter which is equivalent to M_w/M_n as will be understood by those skilled in the art. Such high melt flow resins can be obtained directly in the polymerization reactor using certain "metallocene" catalysts such as those described, for example, in "Metallocene Catalyzed Polymers", edited by G. M. Benedikt and B. L. Goodall, Society of Plastics Engineers, Plastics Design Library series, 1998 or by "cracking" or "vis-breaking" a low melt flow reactor product which is normally required for polypropylene resins produced using Zeigler-Natta catalysts. The cracking or vis-breaking of polymers is well known and involves thermally and/or chemically degrading the polymers to obtain a lower molecular weight product. Representative processes for cracking polyolefin resins, including polypropylene, are described in US Pat. Nos. 3,144,436; 3,887,534; 4,535,125 and 5,587,434.

High melt flow polypropylene resins useful in the compositions of the present invention are advantageously prepared by contacting a low melt flow propylene polymer with an effective amount of an organic peroxide at elevated temperatures in an extruder. A peroxide particularly useful in the cracking of polypropylene resins is 2,5-dimethyl-2,5 bis(t-butylperoxy)hexane. Other peroxides known in the art could also be used. The amount of peroxide used and the cracking temperature will depend upon the melt flow of the starting polymer and the desired melt flow of the final product. Typically, the amount of peroxide used will range between 25 ppm and 5000 ppm. Temperatures in the extruder may range between 180° C and 320°C.

The selected N,N-dialkylhydroxylamines which are part of the compositions of the present invention are known in the art as secondary antioxidant additives useful for stabilizing polypropylene during melt processing. Many specific examples of N,N-

dialkylhydroxylamines useful in the present invention and methods of preparation are specifically disclosed in U.S. Pat. Nos. 4,590,231; 4,782,105; 4,876,300 and 5,013,510, the disclosures of which are incorporated herein by reference. Particularly useful in the compositions of this invention are the long chain N,N-dialkylhydroxylamines disclosed in U.S. Pat. No. 4,876,300 and having the formula



wherein R1 and R2 are independently alkyl groups having 12 to 18 carbon atoms. The most preferred alkyl groups for R1 and R2 are the alkyl mixture found in hydrogenated tallow amine. The amount of N,N-dialkylhydroxylamine incorporated into the compositions of the present invention to achieve the desired results will be in the range of 50 ppm to 5000 ppm based on the total composition and preferably in the range of 200 ppm to 1000 ppm.

The selected organic phosphites and phosphonites which are part of the compositions of the present invention are known in the art as being useful as secondary antioxidants and stabilizers for polypropylene resins and are selected from the group consisting of:

Tris(2,4-di-tert-butylphenyl) phosphite (IRGAFOS 168);
2,4,6 tri-t-butylphenyl 2 butyl 2 ethyl 1,3 propanediol phosphite (ULTRANOX 641);
2,2',2''-nitriolo triethyl-tris[3,3',5,5'-tetra-tert-butyl-1,1'-biphenyl-2,2'-diyl] phosphite (IRGAFOS 12);
Bis (2,4-dicumylphenyl) pentaerythritol diphosphite (DOVERPHOS S-9228); and
Tetrakis (2,4-di-tert-butylphenyl) - 4,4' - biphenylene diphosphite (SANDOSTAB P-EPQ).

Tris(2,4-di-tert-butylphenyl) phosphite is particularly useful and preferred as the organic phosphite which is part of the compositions of the present invention. The amount of organic phosphite or phosphonite incorporated into the compositions of the present invention should be in

the range of 100 ppm to 5000 ppm, preferably in the range of 500 ppm to 2000 ppm.

The compositions of the invention also may contain additives which may be generally termed stabilizers, antioxidants, lubricants, acid acceptors, anti-static agents, nucleating additives and additives which stabilize against radiation, such as ultraviolet (UV) stabilizers and those that provide resistance to gamma irradiation.

Antioxidants which may be most useful in the compositions of the present invention include primary antioxidants of the phenolic-type. Their main function is to provide long-term thermal stability which is usually required in fabricated articles such as fibers and films. Preferred phenolic primary antioxidants include 1,3,5-tris-(4-tert-butyl-3-hydroxy-2,6-dimethylbenzyl) 1,3,5-triazine-2,4,6-(1H,3H,5H)-trione and tetrakis[methylene (3,5-di-tert-butyl-4-hydroxyhydrocinnamate)] methane. Other useful antioxidants include 1,3,5-trimethyl-2,4,6-tris(3,5-di-tert-butyl-4-hydroxy-benzyl) benzene; octadecyl 3-(3',5'-di-tert-butyl-4'-hydroxyphenyl) propionate; tris[3,5-di-tert-butyl-4-hydroxybenzyl) isocyanurate; 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid triester with 1,3,5-tris(2-hydroxyethyl)-s-triazine-2,4,6-(1H,3H,5H)-trione; bis-[3,3-bis(4'-hydroxy-3'-tert-butyl-phenyl)-butanoic acid]-glycolester; 2,2'-methylene-bis-(4-methyl-6-tertiary-butylphenol)-terephthalate; 2,2 bis[4-(2-(3,5-di-tert-butyl-4-hydroxyhydrocinnamoyloxy)) ethoxy-phenyl]propane; calcium bis[monoethyl(3,5-di-tert-butyl-4-hydroxybenzyl)phosphonate]; 1,2-bis(3,5-di-tert-butyl-4-hydroxyhydrocinnamoyl)hydrazine; and 2,2-oxamido bis[ethyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate].

Primary antioxidants such as those specified above may advantageously be combined with secondary antioxidants such as organic phosphites or phosphonites to provide the desired long term thermal stability for the compositions of this invention. To achieve the best results, it is desirable to keep the total amount of primary and secondary antioxidants present under about 1.5% by weight of the composition.

Lubricants or mold release agents are typified by fatty acid amides, examples of which include ethylene bis stearamide, oleamide and erucamide.

Acid acceptors may be categorized as salts of fatty acid, lactic acid salts and related derivatives, hydrotalcite-like compounds, and certain metal oxides. Examples of each type in order include calcium stearate, calcium lactate, DHT-4A, and zinc or magnesium oxide.

Anti-static agents enhance static decay on molded parts. Key examples include glyceryl monostearate and glyceryl distearate, as well as mixtures thereof.

Nucleating additives are typified by benzoic acid salts such as sodium, lithium or aluminum benzoate, minerals such as talc, and organophosphorous salts such as NA-11 and MARK 2180.

Ultraviolet stabilization is provided by light absorbers such as TINUVIN 327 or by hindered amine types such as CYASORB 3346, TINUVIN 622, TINUVIN 770 DF and CHIMASSORB 944.

Resistance against gamma irradiation is provided by combinations of additives such as phosphorous containing secondary antioxidants or the lactone type (e.g. HP-136), and hindered amines. Additionally, Milliken's RS 200 additive is of benefit, as are mobilizing additives such as mineral oil (cited in U.S. Patents Nos. 4,110,185 and 4,274,932). The latter is used in combination with a non-phenolic secondary antioxidant and a hindered amine.

The various types of additives discussed above may be used separately or blended with the primary antioxidants. This applies to all the above additive types and further includes fillers like barium sulfate, clays, calcium carbonate, silicates, pigments, such as titanium dioxide, zinc oxide, lead chromate, cadmium sulfides, cadmium selenide, zinc sulfide, basic carbonate of white lead; stabilizers such as tribasic lead sulfate, basic lead chlorosilicate, dibutyl tin oxide and other salts of lead, zinc, cadmium, tin, and the like; flame retardants such as antimony oxide; ultra-violet stabilizers, slip agents, anti-block

agents, and other solid additives which enhance the properties and processability of the polymer to which they are added.

While the above listing seeks to provide key examples of the different additive types, it is not to be viewed as limited by the examples in scope. It is also recognized that certain of the above additives are multi-functional, e.g., an acid acceptor such as calcium stearate may also provide mold release performance, as may also be the case with glyceryl monostearate. Further, combinations of any or all types of additives given, or of additives within a given class, are considered to be within the scope of the present invention.

Any additives, including the N,N-dialkylhydroxylamine and organic phosphite or phosphonite compounds which are part of the compositions of the present invention may be incorporated into the high melt flow polypropylene resin by conventional techniques at any convenient stage prior to or during the extrusion of the compositions into shaped articles such as fibers or films. For example, the additives may be mixed with the polypropylene resin in dry powder form or as a solution or suspension in an extruder prior to or during the process of cracking the resin or the process of pelletizing the resin.

The effectiveness of the N,N-dialkylhydroxylamine and organic phosphite or phosphonite additive system in reducing smoke may be enhanced by employing the technique of "inerting" in the equipment used to extrude the novel polypropylene compositions of this invention and/or in the area surrounding such equipment. This technique involves maintaining an inert gas atmosphere such as nitrogen or carbon dioxide in the extrusion equipment including the extruder and die and/or in an enclosed space surrounding such equipment.

The following examples are intended to illustrate the present invention but should not be construed as in any way as limiting the scope of the invention. The amount of smoke generated in the process of extruding several polypropylene compositions was measured and compared. The polypropylene resin component was the same in each composition tested and is identified as a polypropylene

homopolymer having a melt flow (MF) of 38 dg/min which was obtained by peroxide cracking a nominal 3 MF polypropylene homopolymer produced in a gas phase reactor using a Zeigler-Natta catalyst. The amount of various additives present in each composition shown in Table I below is given in parts per million (ppm) by weight.

To conduct the smoke measurement tests, the polypropylene resin composition to be tested was fed to a 5 inch slit film die with a 0.020 inch die gap using a Brabender 3/4 inch single screw extruder. The die was completely surrounded by an enclosure having an exhaust chimney at the top which was connected to an exhaust blower. The open bottom of the enclosure through which the polymer extrudate exits was attached to a fabric enclosure which surrounds the extrudate for a distance of 3 feet. With this arrangement, the atmosphere surrounding the die is continuously withdrawn through the exhaust blower. The amount of smoke generated in the atmosphere surrounding the die during the extrusion of each formulation tested was measured using a model HAM-1010 particle detector manufactured by PPM, Inc. This particle detector, which works on a light scattering principal, was connected by sampling tubing to the exhaust chimney which is attached to the die enclosure. A flow meter and vacuum pump were connected to the particle detector.

Various process parameters such as melt temperature, air flow through the particle detector, extruder throughput and air flow past the die were held constant to the extent possible during the testing of all of the various polypropylene compositions. In each test, the polypropylene composition was extruded through the slit die at a throughput of approximately 2.25 kg/hr. The extrudate melt temperature, measured with a hand held pyrometer near the die exit, was 250°C. The exhaust blower was set to provide an air flow through the exhaust chimney of approximately 50 liters per minute (lpm). The vacuum pump and flow meter were adjusted to provide a flow of approximately 1.5 lpm through the particle detector. Sufficient time was allowed for the system to reach steady state conditions before

commencing the collection of data. For each test run, the output of the particle detector was recorded for 20 minutes. The recorded data was then analyzed to determine an "average smoke density" expressed as mg (smoke) per cubic meter of air.

To make a fair comparison of the smoke generated during the extrusion of the various polypropylene compositions tested, three 20 minute runs were made for each composition. The "average smoke density" was determined for each run from the data collected by the particle detector and then the average of the three runs was calculated and used as the "average smoke density" for that composition.

A Control average smoke density value was calculated and used to determine the Smoke Indicator number for the compositions tested which appears in Table I. The Control average smoke density value was determined by calculating the average of the average smoke densities of the first three compositions in Table I. The Control average smoke density value thus represents the average of the average smoke densities for the 9 test runs involved in Examples 1-3 in Table I. The compositions in Examples 1-3 are essentially the same and involve the presence of a known phenolic antioxidant process stabilizer, Irganox 3114 sold by Ciba. The Smoke Indicator number is calculated by dividing the average smoke density of the polypropylene composition of each Example by the Control average smoke density. The lower the Smoke Indicator number, the more resistant the composition is to the generation of smoke during melt extrusion.

Nine polypropylene compositions were tested by the procedure described above and their Smoke Indicator number was determined. The additive content and the Smoke Indicator number for each composition are presented as Examples 1-9 in Table I below.

Table I

ppm

<u>Example</u>	<u>ZnO</u>	<u>CaSt</u>	<u>P1240</u>	<u>3114</u>	<u>1010</u>	<u>168</u>	<u>FS042</u>	<u>Smoke Indicator</u>
1	200	250	-	1000	-	-	-	1.00
2	200	250	-	1000	-	-	-	1.05
3	200	250	-	1000	-	-	-	0.95
4	200	-	-	-	-	1000	500	0.22
5	200	250	-	-	-	1000	500	0.30
6	-	-	400	-	-	1000	500	0.40
7	200	250	-	-	1000	-	-	0.83
8	-	-	400	1000	-	-	-	0.63
9	200	250	-	-	1000	1000	-	1.04

The additives listed in Table I are identified as follows:

ZnO - Zinc oxide

CaSt - Calcium stearate

P 1240 - Calcium lactate

3114 - Irganox 3114 - Ciba - 1,3,5-Tris(3,5-di-tert-butyl-4-hydroxybenzyl)-s-triazine-2,4,6(1H,3H,5H)trione

1010 - Irganox 1010 - Ciba - tetrakis[methylene (3,5-di-tert-butyl-4-hydroxyhydro-cinnamate)] methane

168 - Irgafos 168 - Ciba - tris(2,4-di-tert-butylphenyl) phosphite

FS 042 - Ciba - bis(hydrogenated tallow alkyl) hydroxylamine

As can be readily seen from the Smoke Indicator numbers in Table I, Examples 4-6, which are representative of the high melt flow polypropylene compositions of the present invention, are considerable better than those of the other compositions tested.

What is claimed is:

1. A composition which generates a reduced amount of smoke when melt extruded into a fiber or film which comprises:
 - a) a high melt flow polypropylene resin having a narrow molecular weight distribution and a melt flow of at least about 20 dg/min;
 - (b) an effective amount of an N,N-dialkylhydroxylamine, and
 - (c) an effective amount of an organic phosphite or phosphonite selected from the group consisting of:
tris(2,4-di-tert-butylphenyl) phosphite;
2,4,6 tri-t-butylphenyl 2 butyl 2 ethyl 1,3 propanediol phosphite;
2,2',2''-nitriolo,triethyl-tris[3,3',5,5'-tetra-ter-butyl-1,1'-biphenyl-2,2'-diyl] phosphite;
bis (2,4-dicumylphenyl) pentaerythritol diphosphite; and
tetrakis (2,4-di-tert-butylphenyl)-4,4'-biphenylene diphosphite.
2. The composition of claim 1 wherein said polypropylene resin has a narrow molecular weight distribution M_w/M_n of 2 to 6.
3. The composition of claim 2 wherein the polypropylene resin is obtained by cracking a polypropylene homopolymer, random copolymer or impact copolymer.
4. The composition of claim 1 wherein (b) is a N,N-dialkylhydroxylamine having the formula:



wherein R1 and R2 are the same or different and are alkyl groups containing from 12 to 18 carbon atoms.

5. The composition of claim 4 wherein R1 and R2 are the alkyl mixture found in hydrogenated tallow amine.

6. The composition of claim 5 wherein (c) is tris(2,4-di-tert-butylphenyl) phosphite.

7. The composition of claim 1 which additionally contains:

(d) an effective amount of a primary antioxidant.

8. The composition of claim 7 wherein said primary antioxidant is a phenolic antioxidant or mixture thereof.

9. The composition of claim 1 which additionally contains:

(e) an effective amount of an acid acceptor.

10. A method of reducing the amount of smoke generated during melt extrusion of a high melt flow polypropylene resin to produce a fiber or film which comprises adding to said polypropylene resin prior to or during said melt extrusion:

(a) an effective amount of an N,N-dialkylhydroxylamine, and

(b) an effective amount of an organic phosphite or phosphonite selected from the group consisting of:

tris(2,4-di-tert-butylphenyl) phosphite;

2,4,6 tri-t-butylphenyl 2 butyl 2 ethyl 1,3 propanediol phosphite;

2,2',2''-nitriolo triethyl-tris[3,3',5,5'-tetra-tert-butyl-1,1'-biphenyl-2,2'-diyl] phosphite;

bis (2,4-dicumylphenyl) pentaerythritol diphosphite; and

tetrakis (2,4-di-tert-butylphenyl)-4,4'-biphenylene diphosphite.

11. The method of claim 10 wherein said melt extrusion is conducted in an inert atmosphere.

12. The method of claim 11 wherein said inert atmosphere is nitrogen.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 99/19605

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C08K5/00 C08L23/10 C08K5/526

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C08K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4 668 727 A (RAVICHANDRAN RAMANATHAN ET AL) 26 May 1987 (1987-05-26) column 2, line 31 - line 36; examples ---	1,10
P,X	EP 0 875 530 A (MONTELL NORTH AMERICA INC) 4 November 1998 (1998-11-04) claims 1,4,7,10; examples ---	1,4,9
X	WO 94 24344 A (CIBA GEIGY AG ;HORSEY DOUGLAS W (US); KING ROSWELL E III (US)) 27 October 1994 (1994-10-27) cited in the application page 8, line 5 - line 9 page 8, paragraph 5; tables page 13 -page 16 --- -/--	1,5-7

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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